Title: CONTROLLED LOW STRENGTH FLOWABLE FILL COMPOSITION WITH IRON CHELATING COMPOUNDS

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This application claims priority to co-pending U.S. Provisional Application Serial No. 60/453,231 filed March 10, 2003.

Background of the Invention

This invention relates to Controlled Low-Strength Mixtures (CLSMs), or flowable back-fills. This class of materials has utility as pipe bedding materials has utility as pipe bedding materials where they are used to both protect the pipe from external agents and internal loads. They have also been used as an erosion barrier in embankments and as a mine fill material. CLSMs typically have strengths of less than 2000 psi and, in cases where removal is contemplated, less than 200 psi for ease of removal. The material should be initially in the form of an easily pumpable, self-leveling slurry. Rapid early strength development (approximately 50-70 psi) is a desirable property and is currently not obtainable with commercial products without the penalty of high strength development at later stages. U.S. Patent No. 5,106,422 discloses Class C Fly ash in a rapid setting flowable backfill composition and method for its use.

However, such existing compositions are based upon the use of either Portland cement or Class C fly ash used individually or in combination as the hydraulic cement component of the CLSM system. Typically these cementitious materials are used at less than 5% by weight in the case of Portland cement or as much as 50% in the case of Class C fly ash with the remainder being some form of aggregate, usually fine sand or soil from the spoil with small amounts of additional rock and gravel or Class F fly ash. Cement-based materials can take days to hydrate, cure, and achieve even a

modest strength of 50 psi which is typically the minimum strength required for a man to walk upon the surface of the bedding material and represents the minimum safe time before the cover fill may be placed. Class C fly ash based systems may take as long as four hours to hydrate, cure, and achieve this strength. In many cases, locally available Class C fly ash is not desirable for use in these types of product due to slow hydration, cure, and set times and low strengths. Strength may be compensated for by the use of additional Class C fly ash but the cost of the additional fly ash may result in cost prohibitive products.

Thus, the system of the present invention minimizes the down time before cover fill may be placed and represents a significant savings of both time and money for the user. Furthermore, the present inventive composition and method allow for control variability in strength and hydration, cure, and set times of a CLSM system utilizing Class C fly which results in a Class C-based flowable fill capable of competing in markets previously inaccessible.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a graphic representation of the effect of set time of Class C mortars with lime.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

Class C fly ash as defined in ASTM C 618 is a coal combustion product that meets particular size requirements and mineralogical specifications. A typical chemical composition for this class of fly ash is as follows:

	Percent by Weight
Silicon dioxide (SiO_2) plus aluminum oxide (Al_2O_3) plus iron oxide (Fe_2O_3), min.	50.0
Sulfur trioxide (SO_3), max.	5.0
Moisture content, max.	3.0
Loss on ignition, max.	6.0

This is a rather broad description for this class of material, and significant variability may exist for materials conforming to this requirement. The variability manifests itself as differences in hydration and set time and strength between several samples of Class C fly ash either from the same or different sources. It has been found that a major factor contributing to variability is the amount of available calcium present in the sample. Additionally, soluble iron content contributes to slow setting times. Furthermore, the addition of small amounts of calcium to Class C fly ash has no deleterious effects upon flowable fill and can accelerate the rate of hydration and cure while minimizing the differences in set time and strength of flowable fill mixtures containing Class C fly ash.

Where soluble iron is present in sufficient quantity and extra calcium alone is inadequate to accelerate the rate of hydration, iron chelating compounds may be added, even in very small amounts, to offset the soluble iron effect. The iron chelating compound may include:

sodium chloride, sodium thiosulfate, triethanolamine, diethanolamine, polyethyleneimine, amino-substituted acrylic monomers or polymers, morpholine and substituted morpholine compounds, urea, guanidine salts, pyrole and pyrole compounds, polyvinylpyrole, imidizole compounds, pyrazoles, pyridine and pyridine compounds (especially ortho alkoxy-

substituted pyridines), amino phenol (especially ortho amino phenol), amino cresol, ortho anisidine, amine acetate surfactants (such as Armac HT and Armac 18D-40 from Akzo Nobel Chemicals), amine oxide surfactants (such as Ammonyx series of surfactants from Stepan Company, Schercamox series of surfactants from Scher Chemicals, Foamox series of surfactants from Alzo, Inc., Chemoxide series of surfactants from Chemron Corp.,), amine surfactants (such as the Armeen and Redicote series of surfactants from Akzo Nobel Chemicals, the Incromine series of surfactants from Croda, Inc., the Tealan series of surfactants from R.I.T.A. Corp.), and mercapto surfactants (such as Burco TME from Burlington Chemicals).

The iron chelating compound may be in quantities in the range of 0.01% or 5.0% by weight. Effective results have been obtained and reasonably should be obtained from chelting agents or compounds selected from the group consisting of an alkanolamine, a polymer of ethyleneimine, a block copolymer containing polyethyleneimine segments, an amino-substituted polymer of acrylic acid, the salt of an amino-substituted polymer of acrylic acid, a carboxyated amine compound, a salt of a carboxyated amine compound, ethylenediaminetetraacetic acid and salts thereof; nitrilotriacetic acid and salts thereof, an amine substituted surfactant, an amine oxide substituted surfactant, and a guanidine salt.

The following examples illustrate the nature of the present invention. Set times were determined when a 0.25" diameter penetrometer needle provided a reading of 200 psi on insertion to a depth of 1.0".

Example 1

Coal Fly ash from Deeley Power Plant, San Antonio, Texas, as obtained and used as received. 50 grams of Class C Fly ash, 250 grams ASTM C 33 graded washed silica sand (Espey Sand, San Antonio, Texas) and 35 mL deionized water were mixed for 1 minute and poured into a 2" cube mold. The set time was determined to be 62 minutes as shown in Table 1 below.

Examples 2-8

Coal Fly ash from Deeley Power Plant, San Antonio, Texas, was obtained and used as received. 50 grams of Class C Fly ash, 250 grams ASTM C 33 graded washed silica sand (Espey Sand, San Antonio, Texas), varying amounts of type S hydrated lime and 35 mL deionized water were mixed for 1 minute and poured into a 2" cube mold. The set time for these examples are shown in Table 1 for the varying amounts of lime.

Examples 9-13

Coal Fly ash from Scherer Power Plant, Atlanta, Georgia, was obtained and used as received. 50 grams of Class C Fly ash, 250 grams ASTM C 33 graded washed silica sand (Espey Sand, San Antonio, Texas), varying amounts of type S hydrated lime and 35 mL deionized water were mixed for 1 minute and poured into a 2" cube mold. The set times for these examples are shown in Table 1 for the varying amounts of lime.

A graphic representation of the effect of set time of these Class C motars with lime of varying amounts is shown in Fig. 1.

Table 1. Set times of Class C Fly ash motar cubes containing varying amounts of type S lime.

Lime (grams)	Example Number	Set Time (minutes) Deeley	Example Number	Set Time (minutes) Scherer
0.00	1	62	9	348
0.07	2	51	-	-
0.13	3	26	-	-
0.25	4	12	10	303
0.50	5	10	11	71
0.75	6	8	12	37
1.00	7	12	13	76
1.50	8	9		

Example 14

Ingredient	Amount (g)
Concrete Sand	0
Type C Fly Ash	100
Hydrated Lime	.003
Triethanolamine	.04
Water	25

The dry ingredients were mixed together and the water was added with mixing until a smooth, pourable consistency was obtained. The set time was 17 minutes.

Example 15

Ingredient	Amount (g)
Concrete Sand	200
Type C Fly Ash	100
Hydrated Lime	.3
Triethanolamine	.48
Water	39

The dry ingredients were mixed together and the water was added with mixing until a pourable consistency was obtained. The set time was 17 minutes.

Example 16

Ingredient	Amount (g)
Concrete Sand	250
Type C Fly Ash	50
Hydrated Lime	7.5
Triethanolamine	.1
Water	40

The dry ingredients were mixed together briefly and the water and triethanolamine added with continued mixing. The set time was 23 minutes.